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(54) Title of invention: Aqueous emulsion manufacturing method

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SPECIFICATION

1. [Title of Invention]

Aqueous emulsion manufacturing method

2. [Claims]

1. An aqueous emulsion manufacturing method in which polyalkylene polyamine is reacted in a saturated fatty acid having a carbon number of 12-22, then an inorganic acid, organic acid, or quaternaryizing agent is added and reacted, and with the resulting cationic water-soluble resin as a dispersant, an alkyl ketene dimer is emulsified and dispersed at 50-70°C, then it is given high-pressure emulsifying machine treatment.

2. An aqueous emulsion manufacturing method in which styrene or a styrene derivative and an acrylic monomer are emulsion-polymerized with the cationic water-soluble resin as a dispersant, and with the resulting cationic water-soluble resin emulsion as a dispersant, an alkyl ketene dimer is emulsified and dispersed, then it is given high-pressure emulsifying machine treatment.

3. An aqueous emulsion manufacturing method as described in claim 1 in which an alkyl ketene dimer is emulsified and dispersed, also using in the cationic water-soluble resin a protective colloid selected from the group consisting of a non-ionic or cationic surfactant or polyvinyl alcohol, cationic starch, polyacryl amide, and methyl cellulose.

4. An aqueous emulsion manufacturing method as described in claim 2 in which an alky ketene dimer is emulsified and dispersed, also using in the cationic water-soluble resin emulsion a protective colloid selected from the group consisting of a non-ionic or cationic surfactant or polyvinyl alcohol, cationic starch, polyacryl amide, and methyl cellulose.

3. [Detailed Description of the Invention]

(Industrial field of application)

This invention concerns a method for manufacturing a papermaking sizing agent or a waterproofing agent for cement, gypsum, etc. in which an alkyl ketene dimer is emulsified and dispersed, with cationic water-soluble resin as a dispersant.

(Prior art)

In the papermaking industry, neutral sizing agents have long been used, with calcium carbonate as the filler.

And in recent years calcium carbonate has also been used as a pigment in color-coat paper, and the calcium carbonate content of waste paper has been increasing. Meanwhile, more ground pulp has come to be used as the pulp raw material for papermaking. Neutral sizing agents have had the drawback that if ground pulp is used, no sizing property at all can be expected.

And when it comes to AKD (alkyl ketene dimer) neutral sizing agents in particular, there has been the disadvantage that the startup sizing property is difficult to obtain.

What has been wanted in the papermaking industry is the effect of calcium carbonate, or the manifestation of sizing performance with ground pulp or the appearance of a sizing agent with good startup sizing property.

The inventors of this invention, who have conducted much diligent research to answer these needs of the industry, now make bold to offer this fruit of their efforts.

(Means of solving the problems)

This invention offers an aqueous emulsion manufacturing method in which polyalkylene polyamine is reacted in a saturated fatty acid having a carbon number of 12-22, then an inorganic acid, organic acid, or quaternaryizing agent is added and reacted, and with the resulting cationic water-soluble resin as a dispersant, an alkyl ketene dimer is emulsified and dispersed at 50-70°C, then it is given high-pressure emulsifying machine treatment. It is also possible to use the cationic water-soluble resin as a dispersant to emulsion-polymerize a styrene monomer and acrylic monomer and make the resulting cationic water-soluble resin emulsion into an alkyl ketene dimer dispersant. And the cationic water-soluble resin and the cationic water-soluble resin emulsion can be made into a sizing agent for papermaking, etc., by also using with them a cationic surfactant or non-ionic surfactant such as the high-grade fatty acid trimethyl chloride and emulsifying and dispersing an alkyl ketene dimer.

Any saturated fatty acid of carbon number C12 to C22 can be used in this invention, but it is preferable to use stearic acid. As the polyalkylene polyamide, one can use triethylene tetraamine, diethylene triamine, and tetraethylene pentaamine, but it is preferable to use tetraethylene pentaamine and triethylene tetraamine. For the reaction of the high-grade saturated fatty acid and polyalkylene polyamine, dehydration reactions are carried out by heating and melting at a temperature of 150-250°C, and preferably 180-220°C. The resulting amide is reacted by adding hot water at 50-95°C, and preferably an inorganic acid, organic acid or quaternaryizing agent at 60-80°C, so as to obtain a water-soluble cationic resin.

Also, using the resulting cationic resin as an emulsifier, emulsion polymerization is carried out on styrene, a styrene derivative, and an acrylic monomer to obtain a cationic water-soluble resin.

As the polymerizable monomer to be used here, one may use styrene, α-methyl styrene, acrylamide, acrylnitryl, acryl(methacryl)ate ester, or isobutyl acryl(methacryl)ate, etc.

Also, if necessary for these cationic water-soluble resins, a non-ionic surfactant may be used, but a general polyoxyethylene nonyl phenol ether, polyoxyethylene alkyl ether, or polyoxyethylene sorbitan alkyl ester, etc. is used.

Using the thus-obtained water-soluble cationic resin as a dispersant, it is used 10-90%, and preferably 20-50% on AKD, and after the AKD is dispersed, preliminary dispersal is carried out at 50-80°C, and preferably at 60-70°C, following which a high-pressure emulsifying machine is used, passing it through twice at a pressure of 100-350 kg/cm^2, and preferably 200-300 kg/cm^2, after which it is rapidly cooled to obtain the desired cationic sizing agent for papermaking.

With the resulting cationic sizing agent, poor manifestation of starting degree of sizing and poor manifestation of the degree of sizing when using ground pulp, which were the biggest disadvantages of the previous AKD sizing agent, have been greatly improved, and we are confident that we have provided an answer for what has been a longstanding challenge facing the papermaking industry.

The details are explained in, but are not limited to, the following working examples.

Working example 1

Into a reaction vessel were put 284 parts stearic acid, it was heated and melted, the temperature within the system was brought to 150-160°C, 146 parts triethylene tetraamine were added, and reactions took place for 3 hours at 180°C.

Upon completion of the reactions, the temperature inside the system was brought to 120°C, 1648 parts of warm water (60°C) were added, and the solid portion was adjusted to 20%.

The resulting solution is called solution A.

Next, 800 parts solution A were put into a reaction vessel, the temperature within the system was adjusted to 60-65°C, 35.8 parts epichlorohydrine were added, 143 parts water were additionally added, and reactions were carried out for 5 hours at 65-67°C.

The resulting solution is called solution B.

Next, 150 parts solution B, 70 parts alkyl ketene dimer, and 280 parts water were added to the emulsification vessel, and after dissolving at 70°C, using a piston-type high-pressure emulsifying machine, it was passed through twice at a pressure of 300 kg/cm^2, then it was cooled to 40°C, and its concentration was adjusted to produce a cationic sizing agent of 20% solid content.

Working example 2

Into the emulsification vessel were put 250 parts solution B obtained in working example 1, 200 parts water were added, the temperature inside the system was adjusted to 70 °C, then 1.0 parts α,α-azo-bis-isobutyronitryl was dissolved in a mixed solution of 40 parts styrene and 10 parts isobutyl methacrylate, it was dripped taking 1 hour, and reactions were allowed to take place for 3 hours at 75-80°C.

The resulting polymer material is called solution C.

Next, 250 parts solution C and 200 parts water were put into an emulsification vessel, 50 parts alkyl ketene dimer were further added, it was dissolved and dispersed at 70°C, then, using a piston-type high-pressure emulsifying machine, it was passed through once at a pressure of 350 kg/cm^2, and was cooled and concentration-adjusted to produce a cationic sizing agent of 20% solid content.

Performance tests
Pulp: magazine waste paper (containing 6% $CaCO_3$ content)
Knock-apart degree: CSF [Canadian Standard Freeness] = 330 ml
Basis weight: 100 g/m^2
Quantity of sizing agent added: 0.3%, 0.5%, 0.7% solid content
Fixing pH: adjusted to pH = 6.0 in the sulfuric acid band
Drying conditions: for 2 minutes at 90°C
5' 5 5'
Papermaking steps: dissolving - banding - sizing - papermaking
Papermaking temperature: 40°C
Measurement of degree of sizing: JIS-P-8122 (Steckigt method)

Quantity added Sample	0.3%	0.5%	0.7%
Working example - (1)	18"5	26"1	38"9
- (2)	19"0	30"3	41"4
Commercial AKD sizing agent	0"	1"5	4"8

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